

for 30 minutes represents more than 50% of the weight of the silica present in the suspension, comprising the steps of:

(A) precipitating silica by reacting an acidifying agent with an alkali metal (M) silicate, by:

(i) providing an initial base stock, comprising a proportion of the total amount of the alkali metal silicate introduced into the reaction, the silicate concentration expressed as  $\text{SiO}_2$  in said base stock being lower than 20 g/l,

(ii) adding said acidifying agent to said initial base stock until at least 5 % of the amount of  $\text{M}_2\text{O}$  present in said initial base stock is neutralized,

(iii) adding said acidifying agent to the reaction mixture simultaneously with the remaining amount of alkali metal silicate such that the ratio (amount of silica added)/(amount of silica present in the initial base stock) is between 10 and 100;

(B) separating from the reaction mixture a precipitation cake which has a solids content of between 10 and 40%; and

(C) deagglomerating the said cake to obtain a suspension of low viscosity and wherein said deagglomerating is conducted under conditions that result in a silica suspension which has a stability such that the amount of silica in the supernatant obtained after centrifuging said suspension at 7500 revolutions per minute for 30 minutes represents more than 50% of the weight of the silica initially present in the suspension.

**REMARKS**

Entry of the foregoing amendment, reconsideration and reexamination of the subject application, as amended, pursuant to and consistent with 37 C.F.R. §1.112, and in light of the remarks which follow, are respectfully requested.

By the foregoing amendment, claim 31 has been amended to replace "affected" with "conducted" in part (C) of the claim. This amendment is made in order to address the Examiner's concerns regarding the appropriateness of the word "affected" and is consistent with the wording of claim 39.

Claims 22-37 and 39-45 stand rejected under 35 U.S.C. §103(a) as being allegedly obvious over Chevallier et al (U.S. Patent No. 5,403,570).

The present invention relates to an aqueous silica suspension and a method of making the suspension having specific characteristics. As set forth, e.g., in independent claims 22, 31 and 39, the suspension comprises between 10 and 40% by weight solids, has a viscosity lower than  $4 \times 10^{-2}$  Pa.s at a shear rate of  $50 \text{ s}^{-1}$  and has a stability such that the amount of silica in the supernatant obtained after centrifuging said suspension at 7500 revolutions per minute for 30 minutes represents more than 50% of the weight of the silica initially present in the suspension. The method of making the claimed suspension comprises the steps of:

- (A) precipitating silica by reacting an acidifying agent with an alkali metal (M) silicate, by:
  - (i) providing an initial base stock, comprising a proportion of the total amount of the alkali metal silicate introduced into the reaction, the silicate concentration expressed as SiO<sub>2</sub> in said base stock being lower than 20 g/l,

- (ii) adding said acidifying agent to said initial base stock until at least 5 % of the amount of M<sub>2</sub>O present in said initial base stock is neutralized,
  - (iii) adding said acidifying agent to the reaction mixture simultaneously with the remaining amount of alkali metal silicate such that the ratio (amount of silica added)/(amount of silica present in the initial base stock) is between 10 and 100;
- (B) separating from the reaction mixture a precipitation cake which has a solids content of between 10 and 40%; and
- (C) deagglomerating the said cake to obtain a suspension of low viscosity and wherein said deagglomerating is conducted under conditions that result in a silica suspension which has a stability such that the amount of silica in the supernatant obtained after centrifuging said suspension at 7500 revolutions per minute for 30 minutes represents more than 50% of the weight of the silica initially present in the suspension.

Chevallier et al relates to dispersible precipitated silica particulates and a process for their preparation comprising:

- (a) providing an initial sediment or vessel bottoms which comprises at least a portion of the total amount of silicate required for the reaction and an electrolyte, the concentration of silica in said initial sediment being less than 100 g/l and the concentration of electrolyte in said initial sediment being less than 17 g/l;
- (b) adding the acidifying or acid agent to said sediment until the pH of the reaction medium has attained a value of at least about 7;

- (c) adding additional acidifying agent to the reaction medium and, if appropriate, the remainder of the silicate simultaneously, whereby a suspension is produced in which the maximum proportion of dry solids is 24% by weight; and
- (d) drying the suspension thus produced.

Chevallier et al further discloses that the resulting cake is then subjected to a known disintegration operation such as transferring the cake into a colloidal or ball-type mill (column 4, lines 57-65). The final product produced is a silica powder, preferably having a mean particle size of from 5-70 microns (column 4, line 66 et seq) which is suitable for use in reinforcing elastomer/rubber matrices.

The present claims are not *prima facie* obvious over Chevallier et al at least for the reason that each and every feature of the claims is not disclosed or suggested. For example, the claims include the feature wherein the aqueous silica suspension comprises between 10 and 40% by weight solids, has a viscosity lower than  $4 \times 10^{-2}$  Pa.s at a shear rate of  $50\text{ s}^{-1}$  and has a stability such that the amount of silica in the supernatant obtained after centrifuging said suspension at 7500 revolutions per minute for 30 minutes represents more than 50% of the weight of the silica initially present in the suspension. This feature is not at all disclosed or suggested by Chevallier et al.

In the Official Action mailed May 22, 2000, it is asserted however that although Chevallier et al "does not discuss the features recited in the claims, it [the suspension] is deemed to possess them nonetheless since the steps and conditions [of the process] appear to be the same." Applicants respectfully but strongly disagree with this conclusion of inherency for at least the following reasons.

Simply put, there is no basis to conclude that merely because Chevallier et al may also be concerned with a process of making precipitated silica that the same specific aqueous suspension characteristics would necessarily or certainly be inherent in the suspension of Chevallier et al.

"In relying upon the theory of inherency, the examiner must provide a basis in fact and/or technical reasoning to reasonably support the determination that the allegedly inherent characteristic necessarily flows from the teachings of the applied prior art." *Ex parte Levy*, 17 USPQ2d 1461 (BPAI 1990). The possibility that something may inherently have the characteristics of a claimed product is not sufficient. *Ex parte Skinner*, 2 USPQ2d 1788 (BPAI 1986). The fact that a certain result or characteristic may occur or be present in the prior art is not sufficient to establish the inherency of that result or characteristic. *In re Rijckaert*, 9 F.3d 1531, 1534, 28 USPQ2d 1955, 1957 (Fed. Cir. 1993). "To establish inherency, the extrinsic evidence 'must make clear that the missing descriptive matter is necessarily present in the thing described in the reference, and that it would be so recognized by persons of ordinary skill. Inherency, however, may not be established by probabilities or possibilities. The mere fact that a certain thing may result from a given set of circumstances is not sufficient.'" *In re Robertson*, 169 F.3d 743, 745, 49 USPQ2d 1949, 1950-51 (Fed. Cir. 1999)(citations omitted). See also MPEP §2112.

In the present case, the mere possibility that the suspension described in Chevallier et al may have certain product characteristics or process features in common with the present invention as claimed is not a sufficient basis to conclude with any certainty that it would necessarily have the specific suspension characteristics of Applicants' claims. As such, there is

no reasonable basis to establish and make clear to one of ordinary skill in the art that the features of Applicants' claimed invention which are not disclosed in Chevallier et al would necessarily be present.

In the Official Action, it is further asserted that the steps and conditions of Chevallier et al "appear to be the same" as Applicants' process. However, as has been previously noted in some detail, Chevallier et al does not disclose or suggest the same process since the same steps are not utilized to produce Applicants' suspension. For example, the process of Chevallier et al includes, after step (c) noted above (i.e. adding additional acidifying agent to the reaction medium and, if appropriate, the remainder of the silicate simultaneously), the recovery of the precipitated silica as a filter cake (column 4, lines 15-23) followed by subjecting the cake to "a known disintegrating operation" which "may comprise transferring the cake into a colloidal or ball-type mill" so that the silica may then be spray dried to produce the final silica powder (see especially column 4, lines 57-59 and 53-56). In comparison, Applicant's process includes the step of deagglomerating the precipitation cake to produce an aqueous suspension of precipitated silica having a viscosity lower than  $4 \times 10^{-2}$  Pa.s at a shear rate of  $50\text{ s}^{-1}$  and a stability such that the amount of silica in the supernatant obtained after centrifuging the suspension at 7500 revolutions per minute for 30 minutes represents more than 50% of the weight of the silica initially present in the suspension. This step is not the same as the disintegration step referred to by Chevallier et al at least for the reason that there is no indication that such a disintegration step would necessarily produce a suspension having the features of Applicants' claimed invention.

Further, Chevallier et al utilizes the disintegration step to simply put the filter cake "in a condition which would permit it to be sprayed" (column 4, lines 53-55). This brief amount of information does not provide any basis whatsoever to conclude that by putting the suspension in a condition which allows it to be sprayed would necessarily or certainly produce a suspension having the specific characteristics according to Applicants' claims. As well, for reasons noted above, a mere possibility that such a disintegration step may produce a suspension which might be similar to Applicants' suspension is also not a sufficient basis to conclude that these specific characteristics are inherent.

As such, Applicant's process as claimed is different from the process of Chevallier et al. There is therefore no reasonable basis to conclude that the claimed features of Applicant's suspension would necessarily and certainly be inherent in the suspension of Chevallier et al.

In the Official Action, it is nonetheless asserted that there is "no patentable distinction in 'deagglomerating' versus 'disintegrating'." Applicants respectfully but strenuously disagree.

There is no basis to simply conclude that "no patentable distinction" exists between these two processing steps. No legal or technical support has been provided in the Official Action for this conclusion. It is not proper to simply make categorical assertions without providing any reason why these two steps would not be distinguished.

The information of record further clearly shows that these methods are readily distinguishable. For example, Chevallier et al clearly distinguishes between a known "disintegrating" operation, as it is described at column 4, lines 57-65, to treat the filter cake in order to make the suspension sprayable, and a deagglomeration procedure. Specifically, at column 11, Chevallier et al describes an "ultrasonic deagglomeration" test procedure which is

used to determine the capacity for deagglomeration of the precipitated silica. This deagglomeration test procedure is conducted on a small sample of a 4% aqueous silica suspension (which has been homogenized for two minutes by magnetic agitation) by subjecting the sample to ultrasonic deagglomeration for 420 seconds (7 minutes). The particle size of the silica in the homogenized suspension is then measured using laser diffraction to provide a measure of the capacity of the silica for deagglomeration. As such, a "disintegrating operation," which is used to make the filter cake sprayable so that the precipitated silica may be dried, is different, and therefore readily distinguished from, a "deagglomeration" step at least for the reason that different equipment and procedures are utilized and different objectives are to be met.

Moreover, Chevallier et al does not at all equate or even remotely suggest the use of such a deagglomeration procedure as a known "disintegrating operation" step for the preparation of the dry silica powder. Instead, as noted in the Official Action, the only reference to such known "disintegrating operation" methods is the use of a colloidal or ball-type mill (see column 4, lines 57-59). Clearly, the deagglomeration test procedure utilized to assess the capacity of the silica for deagglomeration is not the same as the use of a colloidal or ball-type mill to disintegrate and dry the filter cake.

In addition, Applicants have previously submitted comparative test results in the Rule 132 Declaration filed March 15, 2000 which establish that the claimed suspension characteristics are not inherent and are not obvious over Chevallier et al. In particular, these results provide a comparison of the effects of increasing deagglomeration times by ultrasonic deagglomeration of five Si/Al cake samples prepared according to example 2 of the instant application. These samples were prepared according to steps (A) and (B) of the invention, and also according to the

method disclosed in Chevallier et al. Each cake was then subjected to chemical crumbling and ultrasonic deagglomeration as described at page 4 of the Declaration. As shown in Table I (page 4 of the Declaration), only examples 4 and 5 provide a silica suspension having the specific viscosity and suspension stability characteristics according to Applicants' claimed invention (as measured by the amount of silica in the supernatant following centrifugation). Further, example 1 corresponds to the process disclosed in Chevallier et al and does not possess the claimed viscosity and suspension stability characteristics. Examples 2 and 3 are further treated by deagglomerating for 5 and 10 minutes, respectively, and show that these suspensions also do not possess the claimed characteristics. A deagglomeration time of 7 minutes (420 seconds), as utilized by Chevallier et al in the deagglomeration test procedure described at column 11, does not therefore produce a suspension having the viscosity and suspension stability characteristics of Applicants' claims. Consequently, it is apparent that the claimed suspensions possess viscosity and stability characteristics which are not disclosed or suggested by Chevallier et al.

In the Official Action, a number of objections have been made to the Rule 132 Declaration filed March 15, 2000 (page 3 of the Official Action). Applicants respectfully submit, for at least the reasons noted below, that these objections do not in any way render the Declaration insufficient to overcome the rejection based on Chevallier et al.

For example, under objection 1), the statement that "it is not clear how example 1 could represent the two products of Chevallier example 4" appears to be based upon a misunderstanding of the Declarant's statement. Specifically, it was stated in the Declaration that "Example 1 illustrated the process disclosed in chevallier et al" and that "Examples 4 and 5 illustrated the process of the instant invention." See page 4 of the Declaration after Table I.

These statements do not imply that example 1 (of Chevallier et al) somehow represents the two products of “Chevallier example 4.”

The statement under objection 2) that “the Chevallier example chosen is not representative of the Chevallier product and is therefore not a comparison to the closest prior art” furthermore is not a reasonable characterization of what is shown in the Declaration. Rather, Applicants have shown a direct comparison to a specific example (example 1) from Chevallier et al to establish the novelty and non-obviousness of their claimed method and product. A pertinent comparison to the “closest prior art” has therefore been shown in the Declaration.

With regard to why the “protocol” and “the extra steps were chosen” referred to in objection 3), it is simply noted, as explained in the Declaration, that the results presented demonstrate that the deagglomeration step utilized in Applicants’ invention produces a suspension with the claimed characteristics. This is clearly demonstrated in examples 4 and 5 of the Declaration. On the other hand, other processes of producing a silica suspension, such as according to example 1 of Chevallier et al, or even by ultrasonic deagglomeration of such a silica suspension for five or even ten minutes, do not produce a silica suspension having the claimed characteristics. As clearly emphasized in the Declaration, these results are fully unexpected since the possibility of reagglomeration phenomena in silica suspensions does not obviously lead to a long term stability.

The claims are furthermore commensurate in scope with the showing of results since the specific suspension characteristics claimed are produced by the claimed deagglomeration step. In particular, the claims clearly specify the invention in terms of the viscosity and stability characteristics of the suspension which are produced by the deagglomeration step. Applicants

are furthermore clearly entitled to claim their invention in terms of the effect produced by a method step, namely the specific characteristics of the suspension. There is therefore no need to also claim the deagglomeration time since the result of the deagglomeration step is the specific suspension claimed.

With regard to the statement under objection 5) that “the statement ‘an intensive deagglomeration does not obviously lead to a long term stability’ is incorrect,” Applicants respectfully submit that there is no basis for this conclusion. No evidence or other technical reason has been provided in the Official Action to show that Declarant’s statement is not correct. While it is further stated “one wishing to increase stability *would* find it obvious to deagglomerate as much as possible to prevent reagglomeration . . . [to] disperse as much as possible,” Applicants respectfully submit that this is simply a conclusion or presumption which is unsupported by anything on the record other than Applicants’ own results. Other than these results, there is nothing which establishes that a greater intensity or time of deagglomeration produces a greater suspension stability, let alone that reagglomeration is prevented.

In this regard, Applicants further submit that prior to Applicants’ invention it was not known, and was absolutely not obvious, that an intensive deagglomeration of silica would lead to an improved suspension stability. In particular, it is well known that silica particles dispersed in water tend to reaggregate, e.g. due to Van der Waals type interactions. The production of silica suspensions having high stability has therefore been difficult to predict. As previously noted in the Remarks filed March 15, 2000, this unpredictability in suspension stability is substantiated by French Patent Application 2453880 (U.S. counterpart Patent No. 6,013,234 to Ray et al). As such, the unpredictability of the stability of silica suspensions does not reasonably

allow one to conclude with any certainty that deagglomeration would prevent reagglomeration as alleged in the Official Action, or that the suspension stability would be improved according to Applicants' claimed invention.

Applicants further note that the pH of the filter cake of Chevallier et al is about 7. This pH corresponds to a specific pH domain wherein it is known that silica particles have no surface charges which are able to counteract the attraction effects of Van der Waals' interactions. For example, as shown in the attached copy of Figure 9, page 19 from *The Colloid Chemistry of Silica* (1990), a colloidal silica-water system would be expected to lead to a rapid aggregation at a pH near 7. Such suspensions would therefore not be expected to be stable.

In the Official Action, it is further asserted that Chevallier et al "appears [to] render[s] obvious the claimed steps." Applicants respectfully disagree.

Chevallier et al does not disclose or in any way suggest a deagglomeration step in a process according to Applicants' claimed invention. As well, there is no disclosure or suggestion to add a deagglomeration step to Chevallier et al's process of producing a dry silica powder. Instead, Chevallier et al only discloses that the suspension may be subjected to a disintegrating operation so that it may be spray dried. This suspension, as noted above, is not subjected to a deagglomeration step. Such a deagglomeration step is furthermore not equivalent to a disintegrating operation. While Chevallier et al does measure the capacity for deagglomeration of the dried silica by forming an aqueous suspension and subjecting the suspension to ultrasonic deagglomeration, this test procedure in no way suggests any reason for adding a deagglomeration step to the method of forming the suspension which is to be spray dried. This procedure is only

used as a test procedure, not a "disintegrating operation" (e.g. using a colloid or ball-type mill), and does not provide any motivation to modify the process of Chevallier et al.

Moreover, even if one were (for some unknown reason) to add such a deagglomeration step to Chevallier et al's process of producing a dried silica powder, adding such a step as disclosed in the deagglomeration capacity test procedure at column 11 would still not produce a suspension having the specific suspension characteristics of Applicants' claimed invention. In particular, as shown in the Rule 132 Declaration filed March 15, 2000, the claimed suspension viscosity and stability characteristics are clearly not produced when a deagglomeration time of between five and ten minutes is used. Since Chevallier et al only discloses that the "[d]agglomeration is effected for 420 seconds" and does not at all suggest that longer times should or may be used for any particular reason, there is no motivation to utilize a longer deagglomeration time.

Further, there is no expectation of success in achieving any particular result by utilizing a longer deagglomeration time since Chevallier et al does not in any way disclose or suggest any benefit in varying the deagglomeration time. More specifically, there is no link between the mere possibility of varying the deagglomeration time with any particular result which would be expected to be achieved by adding a deagglomeration step to Chevallier et al's process and also increasing the deagglomeration time to achieve that (unknown) particular result. Simply put, the deagglomeration time has not been shown to be a result-effective parameter based upon Chevallier et al to suggest any motivation to add such a step, nor any expectation of success were such a step to be added, to the process of Chevallier et al. Instead, only Applicants' disclosure provides the information that the suspension stability according to the present claims may be

produced by the claimed deagglomeration step. Clearly, Applicants' disclosure in this regard may not be relied upon as a basis to reject the claims.

Applicants further provide an additional attached Rule 132 Declaration which demonstrates that the deagglomerated "test procedure" suspension of Chevallier et al disclosed at column 11 is not stable according to Applicants' suspension. In particular, the results of this Declaration are comparative trials conducted on suspensions prepared by re-dispersing in water the solid silica powder of Example 1 of Chevallier et al. As explained at page 3 of the Declaration, a 4% aqueous suspension of silica prepared according to Example 1 of Chevallier et al, as well as test samples also produced according to Chevallier et al but having 10% and 20% by weight silica concentrations, respectively, were assessed for the stability of the suspension. In each case, a sedimentation (plug) resulted within 30 minutes. In comparison, three silica suspensions prepared at the same 4%, 10% and 20% by weight concentrations, but according to Applicants' process, did not produce observable settling after 30 minutes. As such, these results demonstrate that Applicants' claimed process produces a stable suspension whereas the suspension of Chevallier et al's test procedure is not stable. As with the results presented in the earlier Rule 132 Declaration of March 15, 2000, these results further confirm that the claimed suspension and process is not obvious over Chevallier et al.

Based on the foregoing, the present claims are patentable over Chevallier et al. Withdrawal of the rejection of Claims 22-37 and 39-45 based on Chevallier et al is respectfully requested.

Claims 38 and 46 stand rejected under 35 U.S.C. §103(a) as being allegedly obvious over Chevallier et al (U.S. Patent No. 5,403,570) as applied against Claims 22-37 and 39-45 in the preceding rejection, and further in view of Cox et al (U.S. Patent No. 4,837,195).

Cox et al relates to a process for porosity control and rehydroxylations of silica bodies by contacting the bodies with an effective amount of ammonium bifluoride. Following the disclosed chemical treatment with ammonium bifluoride, the treated silica may be washed with water or other solvents.

For the reasons noted above and incorporated herein, Chevallier et al does not render Claims 22-37 and 39-45 obvious. Cox et al furthermore fails to cure the deficiencies of Chevallier et al since Cox et al does not disclose or suggest a deagglomeration step to produce Applicants' claimed suspension. Since Claims 38 and 46 depend from independent Claims 31 and 39, they are therefore also patentable over the combination of applied documents for at least these same reasons.

Claims 38 and 46 are further patentable over Cox et al combined with Chevallier et al since these documents are not properly combined. There is no apparent reason to utilize an organic solvent washing step, which may be disclosed as suitable for the ammonium bifluoride reaction process of Cox et al, in a completely different process of treating silica according to Chevallier et al. Merely because a particular treatment step may be known in the art does not suggest a particular and appropriate motivation to add such a feature to the invention of Chevallier et al.

Based on the foregoing, Claims 38 and 46 are patentable over the combination of Chevallier et al and Cox et al. Withdrawal of the rejection of the claims is requested.

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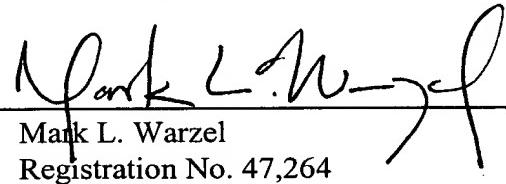
From the foregoing, further and favorable action in the form of a Notice of Allowance is believed to be next in order, and such action is earnestly solicited.

If any issues remain outstanding, the Examiner is respectfully requested to contact the undersigned so that prosecution may be expedited.

Respectfully submitted,

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**Attachment to Reply and Amendment Pursuant to 37 C.F.R. §1.111  
dated March 16, 2001**

**Marked-up Version of Claim 31**

Claim 31 is amended as follows:

31. (three times amended) A method for the preparation of an aqueous suspension of precipitated silica, having a solids content between 10 and 40% by weight, a viscosity lower than  $4 \times 10^{-2}$  Pa.s at a shear rate of  $50\text{ s}^{-1}$  and wherein the amount of silica present in the supernatant obtained after centrifuging said suspension at 7500 revolutions per minute for 30 minutes represents more than 50% of the weight of the silica present in the suspension, comprising the steps of:
- (A) precipitating silica by reacting an acidifying agent with an alkali metal (M) silicate, by:
- (i) providing an initial base stock, comprising a proportion of the total amount of the alkali metal silicate introduced into the reaction, the silicate concentration expressed as  $\text{SiO}_2$  in said base stock being lower than 20 g/l,
- (ii) adding said acidifying agent to said initial base stock until at least 5 % of the amount of  $\text{M}_2\text{O}$  present in said initial base stock is neutralized,
- (iii) adding said acidifying agent to the reaction mixture simultaneously with the remaining amount of alkali metal silicate such that the ratio (amount of silica added)/(amount of silica present in the initial base stock) is between 10 and 100;
- (B) separating from the reaction mixture a precipitation cake which has a solids content of between 10 and 40%; and
- (C) deagglomerating the said cake to obtain a suspension of low viscosity and wherein said deagglomerating is [affected] conducted under conditions that result in a silica suspension which has a stability such that the amount of silica in the supernatant obtained after centrifuging said suspension at 7500 revolutions per minute for 30 minutes represents more than 50% of the weight of the silica initially present in the suspension.